National Bureau of Standards

June 1959
Volume 43
Number 6

Technical News Bulletin

MACHINE TRANSLATION OF RUSSIAN

EXPERIMENTS in machine translation which the Bureau has been performing for the Army Office of Ordnance Research now point the way to practical production of English text from Russian technical literature. A translation scheme recently developed instructs an electronic computer to weld together the English correspondents of the Russian words in a given text to make a meaningful reproduction of the intent of the original sentence. Feasibility studies have been carried out on a high-speed electronic computer by Ida Rhodes of the mathematical staff. The fact that she is a linguist as well as a mathematician and computer programer greatly facilitated the development of the system.

The program first instructs the machine to change the words of a Russian sentence into a highly condensed form for matching in the glossary. The machine is then told to recognize the syntactical relations between the words. After this, it puts together the English words into a meaningful sentence. With the improvements and shortcuts that are expected to be developed in the programing, and by using such advanced computers as are now under construction, the cost may be reduced to no more than a human translator charges.

From a theoretical point of view, the problem of translating from one language into another is not completely solvable. A sentence does not always successfully express a thought; furthermore, it is not possible to be sure that this thought was perfectly formed before it was stated. Yet it is the task of the translator to render such a possibly distorted statement into a target (translated) sentence which conveys the intent and import of the source (original) sentence. With the additional requirement that the task be automatized, the problem becomes quite formidable, especially since present-day computers do not have the capacity for rapid, economical translation. In view of these facts, what has been attempted at the Bureau is experiments in *practical* translation. Such a translation may be neither elegant nor complete, but nevertheless should convey the gist of the source material intelligibly and faithfully.

The method consists of two parts. The first part merely "looks up" every source word in a Russian-English glossary. This glossary is stored, in greatly condensed form, on external media such as magnetic tape. Since Russian is a highly inflected language, only the stems are kept in the glossary. Prefixes and suffixes are also stored, but separately from the glossary. To further conserve limited memory space, instructions dealing with refinements of English grammar are not used. For instance, all words are treated as regular. While this may lead to such inelegant expressions as "good-er" and "good-est", at least the intent is clear.

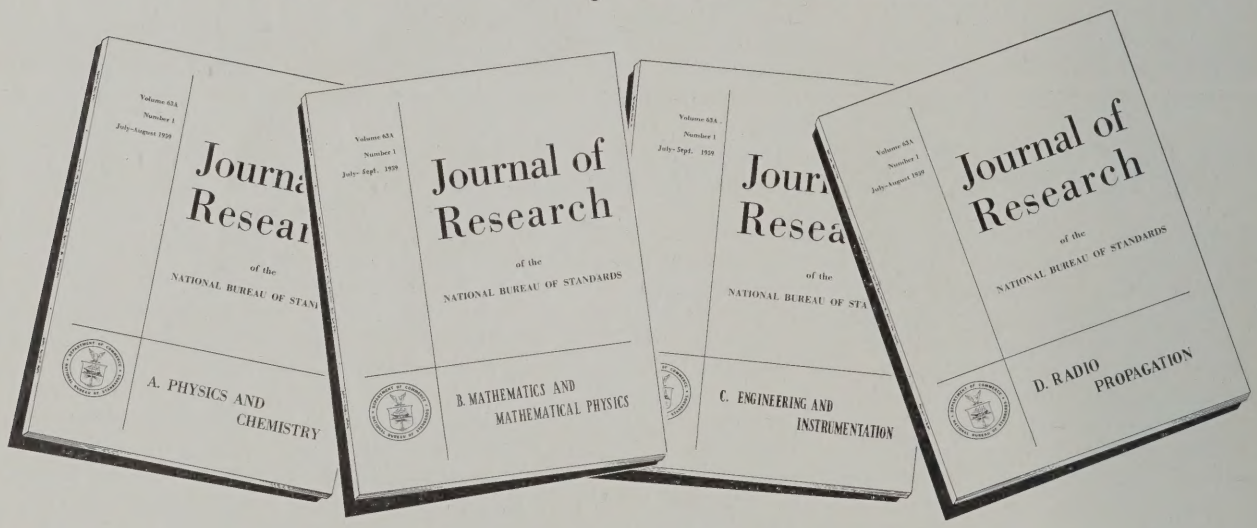
The information obtained from the look-up in part 1, combined with the information for the ending of a source word, is used in part 2 of the scheme to weld together the individual target words into a meaningful, if not esthetic, English sentence. The crux of this iterative scheme lies in the fact that most words of a

language have ascertainable tendencies to associate with other words. These tendencies are listed in the glossary along with the syntactical, grammatical, and lexicological properties of each stem. As each source occurrence is examined, predictions are made—on the basis of these tendencies—concerning the nature of the occurrences to be expected. The scheme progresses by linking together words which seem to fit each other and by carefully recording the doubts, conflicts, and indecisions that may arise. By alternately looking forward to make predictions and backward to ascertain whether light has been thrown upon previous conflicts, the target words are gradually modified and rearranged, in the manner of jigsaw pieces, to complete the puzzle.

A number of criteria serve to indicate whether the current attempt is successful or not. For example, if a prediction which was indexed as a "must" (such as the necessity for a sentence to have a subject, actual or implied) has not materialized, another iteration will be attempted, aided by the facts garnered in the previous ones. The scheme puts a limit on the number of allowable iterations, since it is quite possible to enter upon an endless repetitive process because of the inadequacy of the scheme to cope with all possible situations. In case of failure, a word-to-word translation is printed out with an indication of where the scheme failed. Even when the tests indicate no failures, it is not at all certain that the final sentence is an accurate one. But since the English language is far less sensitive to syntactical and grammatical variations than is the Russian, in the majority of cases the English sentence should be meaningful and faithful. Should it turn out to be otherwise, it should furnish clues as to how the translation scheme might be improved.

Announcing:

New Four-Part Journal of Research



AFTER July 1, 1959, the National Bureau of Standards will begin publishing its *Journal of Research* in four separate sections, corresponding to subject matter fields, which may be subscribed for individually. This change is being made to permit more effective dissemination of the Bureau's findings to science and industry. At the same time the editorial scope of the *Journal* is being broadened to cover the Bureau's technical program as completely as possible. Other current publications changes include the initiation of two new nonperiodical publication series.

The *Journal of Research of the National Bureau of Standards* is the basic medium by which the Bureau reports its findings to the scientific community. The *Journal* contains comprehensive research papers which give complete details of the work, including laboratory data, experimental procedures, and theoretical and mathematical analyses.

As now published, the content of the *Journal* is as broad as the Bureau's scientific program, embracing most fields of the physical sciences as well as various branches of engineering. The division of the *Journal* into separate sections is an effort to meet more effectively the specialized needs of today's scientists, engineers, and mathematicians. Henceforth, the individual reader need subscribe only to those sections of the *Journal* that fall within his particular field of interest.

Four Sections

The *Journal* is being divided into the following sections, each available upon subscription from the Government Printing Office:

Section A: Physics and Chemistry
Section B: Mathematics and Mathematical Physics

Section C: Engineering and Instrumentation
Section D: Radio Propagation

Section A, *Physics and Chemistry*, will cover a broad range of physical and chemical research, with major emphasis on standards of physical measurement, fundamental constants, and properties of matter. It will be issued six times a year, and the annual subscription will cost $4.00 (75 cents additional for foreign mailing).

Section B, *Mathematics and Mathematical Physics*, will present studies and compilations designed mainly for the mathematician and theoretical physicist. It will include topics in mathematical statistics, theory of experiment design, numerical analysis, theoretical physics and chemistry, and logical design and programing of computers. Short numerical tables will also be included from time to time. Section B will be issued quarterly, and the annual subscription cost will be $2.25 (foreign, 50 cents additional).

Section C, *Engineering and Instrumentation*, will report results of interest chiefly to the engineer and the applied scientist. This section will include many of the new developments in instrumentation resulting from the Bureau's work in physical measurement, data processing, and development of test methods. It will also cover some of the work in applied mechanics, properties of engineering materials, building research, and cryogenic engineering. Section C will be issued quarterly, and the annual subscription cost will be $2.25 (foreign, 50 cents additional).

Section D, *Radio Propagation*, is to be edited at the Bureau's Boulder (Colo.) Laboratories. It will report research in radio propagation, communications, and upper atmospheric physics. Topics to be covered include propagation in ionized media, scattering by turbulence, effect of irregular terrain on propagation, diffraction and scattering by solid obstacles, propagation through time-varying media, surface waves, and antennas. Section D will be issued six times a year, and the annual subscription cost will be $4.00 (foreign, 75 cents additional).

Broadened Scope

The editorial scope of the *Journal* will be broadened in several ways. In addition to research reports, the new *Journal* will present review articles by recognized authorities and compilations of information on subjects closely related to the Bureau's basic mission. Much of the material which the Bureau has been publishing in nonperiodical form will henceforth be directed to the *Journal*.

To provide complete coverage of the Bureau's technical program, all NBS nonperiodical publications and articles by the Bureau staff in professional journals will be abstracted in the appropriate section of the *Journal of Research*. In addition, each section will carry a complete listing of all Bureau publications that are not abstracted in that section. Thus, within a particular *Journal* section, each individual Bureau publication will appear either in full, in abstract, or listed with appropriate reference data.

Editorial Appointments

Another change is the appointment of members of the Bureau's technical staff to serve as editors of the various sections, and the appointment of advisers from the Institute of Radio Engineers to Section D, *Radio Propagation*. Those selected are listed below:

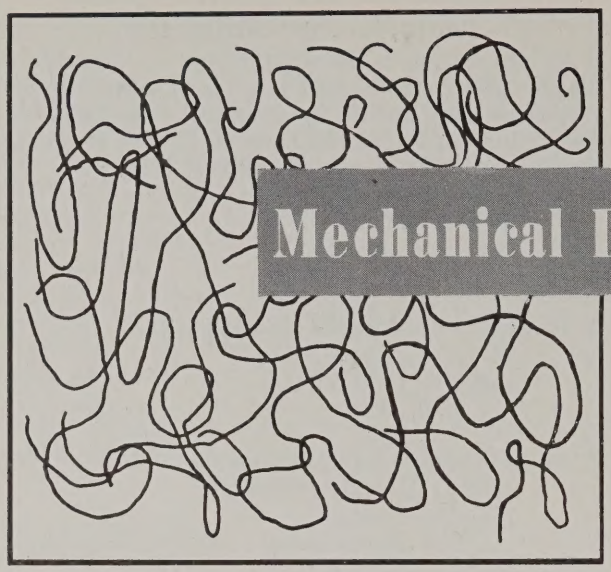
A. Physics and Chemistry—
  Editor: U. Fano
  Associate Editors:
    D. D. Wagman
    J. M. Richardson
B. Mathematics and Mathematical Physics—
  Editor: Chester H. Page
  Associate Editors:
    F. L. Alt
    E. L. Crow
C. Engineering and Instrumentation—
  Editor: William A. Wildhack
  Associate Editors:
    G. F. Montgomery
    R. B. Jacobs
D. Radio Propagation—
  Editor: James R. Wait
  Associate Editors:
    Thomas N. Gautier
    Jack W. Herbstreit
    C. Gordon Little
    Alvin G. McNish
  IRE Advisers:
    D. G. Fink
    K. M. Siegel

Other Publication Changes

Two of the Bureau's nonperiodical series—*Circulars* and *Building Materials and Structures Reports*—are being discontinued inasmuch as much of the material now appearing in these series will go into the new, expanded *Journal of Research*. At the same time, two new nonperiodical series are being inaugurated. These series will be known as *Monographs* and *Technical Notes*. No change is contemplated in the Bureau's three other nonperiodical series (*Applied Mathematics Series, Handbooks,* and *Miscellaneous Publications*) nor in the other NBS periodicals (*Technical News Bulletin* and *Basic Radio Propagation Predictions*).

NBS Monographs will consist of major contributions to the technical literature which are too long for publication in the *Journal of Research*. They will comprise much of the type of material that is now published in the larger circulars such as, for example, *Atomic Energy Levels, Tables of Thermal Properties of Gases,* and *Worldwide Occurrence of Sporadic E*.

NBS Technical Notes have been designed to supplement the Bureau's regular publications program. They will provide a means for making available communications and reports that are of transitory or limited interest. *Technical Notes* will be offered for sale by the Office of Technical Services, U.S. Department of Commerce, Washington 25, D.C. All other NBS publications are available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.



Representation of molecular entanglement. This intertwining of polymer chains is thought to be involved in concentrating sufficient energy into a bond to activate rupture.

Mechanical Degradation of Polymers

TO OBTAIN basic information on the strength of intermolecular entanglement in polymers and their flow stability, the Bureau, in connection with the Government Synthetic Rubber Program, has been investigating the effects of mechanical shearing on polymers in concentrated solutions.

When high-polymer molecules are subjected to severe mechanical shearing, they are sometimes broken down into smaller molecules. This degradation may be produced either by the heat dissipated in the shearing process or directly by the mechanical forces involved in the shearing. Isolating the degradation due to the mechanical forces themselves, A. B. Bestul [1] and coworkers in the rheology laboratory have established several defining features of degradation associated with mechanical shearing. Their research indicates that, under a given set of shearing conditions, a polymer will degrade to a molecular weight level representative of these shearing conditions and below which no further degradation will occur. The occurrence, rate, and ultimate extent of rupture depend on the occurrence and severity of the shearing under otherwise identical conditions. The degradation does not result either from incomplete solution prior to shearing nor from cavitation in the shear field, but is caused by the shearing itself.

Work on degradation is useful both in theoretical and applied areas of polymer science. In the theoretical field, research on mechanical shearing is leading toward better understanding of high-polymer structures, especially of entanglements and other interconnections between such molecules. In the applied field, for such technological processes as lubrication, pumping, ultrasonic applications, milling, shaking, turbulent flow, and lamellar flow, polymeric substances are deliberately or incidentally subjected to shearing—which may be severe enough to cause rupture within the molecule. By studying mechanical degradation, the use of polymers that degrade excessively under severe conditions can be avoided and special polymers that will remain intact under such conditions can be developed.

Procedure

Unfractionated polyisobutene in *n*-hexadecane solution was the polymer system most thoroughly investigated. Solutions containing from 3- to 20-mole percent of the polymer were sheared by being repeatedly forced through the capillary of the McKee consistometer,[2] an instrument developed to test experimental fluids of high viscosity. This device has a capillary hole penetrating a disk which is clamped between two steel cylindrical tubes, each equipped with a closely fitted piston. The pistons repeatedly force the polymeric solutions to pass back and forth through the disk. As the instrument is essentially symmetrical with respect to the cylinders and has pistons on both sides of the disk, successive measurements can be made by moving the pistons in alternate directions.

Records were made of the load on the consistometer for every other pass of a sample. Data were obtained for nominal rates of shear from 9,000 to 100,000 sec^{-1}, temperatures from 20° to 80° C, and initial viscosity-average molecular weights from 600,000 to 2,520,000. Some of the runs were interrupted at various stages of the degradation process. Viscosity-average molecular weights were then determined for the polymers present at that particular stage of shearing, and the extent of degradation determined from these.

To examine specific details of the process, incidental measurements were made on solutions of polymer fractions obtained from degraded and undegraded starting materials and of polymers precipitated completely from degraded stock. In some of these tests, shearing was interrupted for different periods but subsequently completed.

Energy Source

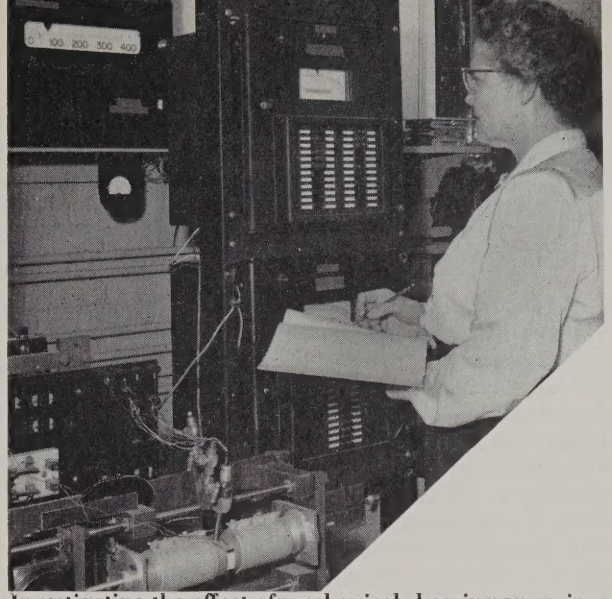
It was found that the rate of degradation is proportional to the concentration of the bonds present at fixed shearing conditions. Within certain conditions—polymer concentrations between 5- and 20-weight percent and temperatures between 30° and 50° C—the proportionality factor between degradation rate and bond concentration (rate constant) is independent of temperature and bears the same relation to the rate of shear energy application that exists between the rate constant and temperature in thermally activated chemical reactions. This similarity suggests that mechanical shear degradation can be considered as a mechanically activated reaction, and leads to a distinguishing feature between the molecular processes occurring in

mechanical and in chemical degradations. This characteristic is the source of energy which raises the energy state of molecular bonds to a level high enough to cause rearrangement of the atoms. In shear degradation, this energy appears to be supplied from mechanical energy associated with the shear field rather than from thermal or electromagnetic sources, for example.

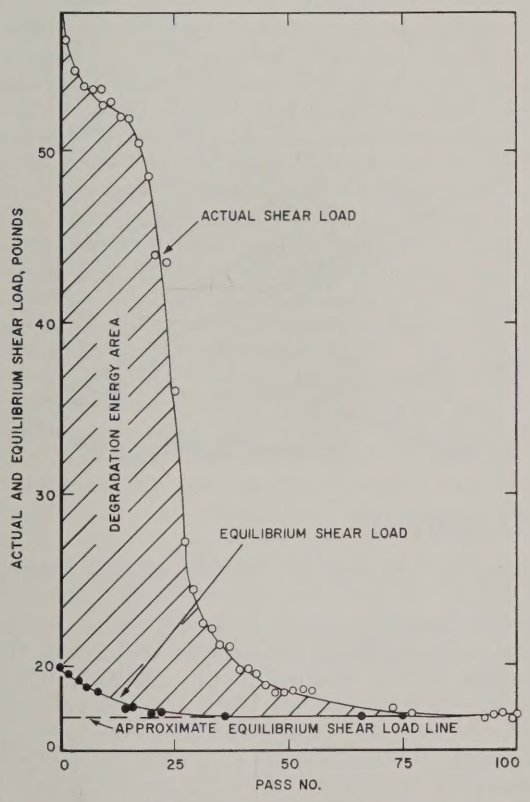
If the energy generated in the shear field is uniformly distributed, the energy developed in the volume occupied by an individual bond (and the two atoms it connects) can provide only a small part of the necessary energy. Therefore, to provide the energy needed to rupture a single bond, energy developed in a larger volume than the immediate bond vicinity is required. The energy from this large volume of the shear field must be in some way concentrated into the bond. It has been suggested that this concentration may be accomplished by the accumulation of stresses due to the shearing through entangled molecular chains, i.e., overlapped or entwined polymers.

Energetics

Another characteristic of mechanical shear degradation is the large amount of energy used by the process per bond ruptured—the values were found by the Bureau to be in the neighborhood of 300,000 kcal/mole of bonds ruptured. Since this large amount of energy is several thousand times any reasonable value for the bond energy of carbon-carbon bonds, it is evident that some of the energy is not utilized in the rupture itself but is somehow lost in the process. This may occur during bond rupture when part of the energy stored in neighboring bonds or macromolecular segments may be immediately dissipated as heat, rather than first being utilized in the shearing process. This energy would be measured and included in the overall energy loss for the degradation process.



Investigating the effect of mechanical shearing on various concentrated polymeric solutions. Here a scientist records the force required to shear polymers in a consistometer.



If no bond rupturing were occurring, the shear forces would be expected to be much lower than observed. Open circles represent the observed shearing force, while closed circles represent an estimate of the shearing forces which would exist for the molecular weight prevailing at various stages of degradation—if no degradation were occurring. Since the difference between the two curves represents that contribution to the shearing forces which is associated with the degradation process, the shaded area represents the amount of degradation energy required.

Although general information on the number of neighboring bonds or macromolecules which are instrumental in transmitting activation energy to a ruptured bond can be estimated, detailed data cannot be obtained, as it is not as yet possible to measure several quantities necessary for detailed calculation. These undetermined values include the actual amount of energy accumulated in individual neighboring bonds, the proportion dissipated as heat immediately upon rupture, and the respective proportions stored as potential energy in the bonds and as reduced configurational entropy of the macromolecular chains formed by the bonds. However, results on fractionated polymers precipitated from unsheared and previously sheared samples support the conclusion that the transmission of activation energy into a single ruptured bond involves several polymeric chain macromolecules. In the degradation, the individual macromolecules with the highest molecular weights are ruptured. But degradation occurs only if at least a minimum concentration of these large molecules is present.

Under extreme conditions (relatively low concentration or high temperature) both the rate and energy requirements of mechanical shear degradation deviate considerably from the above results. That is, the rate varies much more rapidly with the temperature, and

the energy requirements are higher. Presumably the polymer cannot form sufficient effective entanglements at low concentrations and the viscosities of the solutions are too low to develop adequate shearing forces at high temperatures.

Effect of Capillary Geometry

Recent work with consistometer capillaries of different lengths and diameters has indicated that shear degradation occurs in the entrance end of the capillary for a distance many times the capillary diameter. Interesting effects of the exact geometry of the capillaries have been demonstrated [3] with capillaries having exaggerated deviations from right-circular, cylindrical geometry. These variations took the form of a chamfered and a tapered bore at one end of the capillary. The taper has a marked effect on the degradation results, as shown by the substantially different degradation patterns observed in flow through this type of capillary in the two opposite directions. However, the chamfer at the edge of the capillary has little effect on this degradation, presumably because it does not extend over an appreciable part of the capillary distance where degradation occurs.

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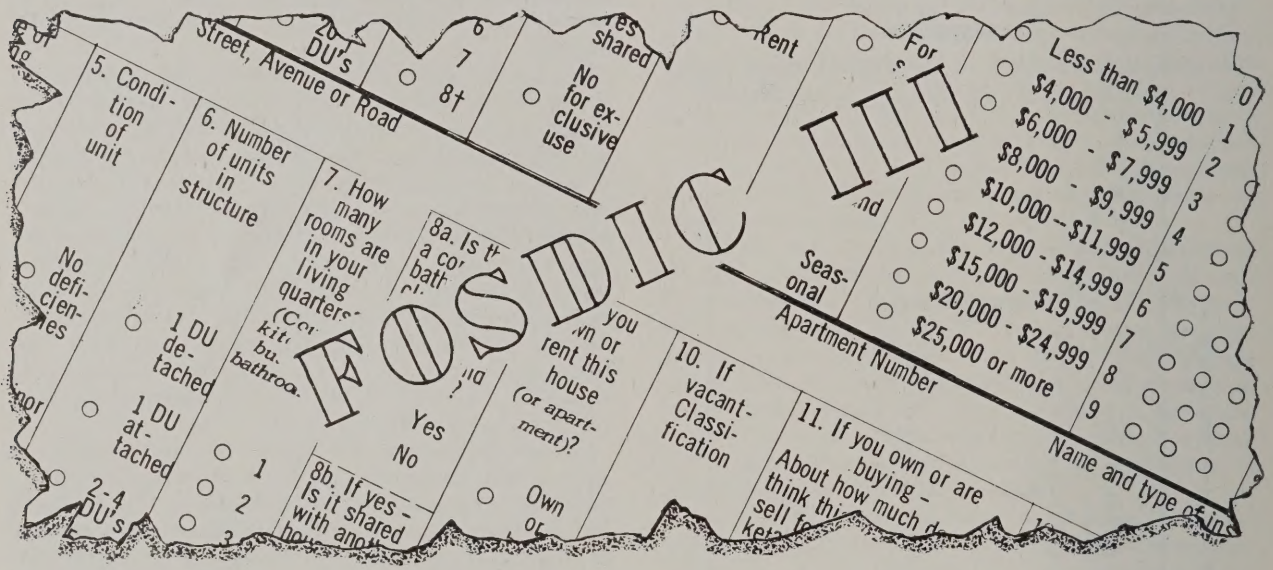
[1] *For further technical details, see* Energy requirements of mechanical shear degradation in concentrated polymer solutions, *A. B. Bestul, J. Chem. Phys. (in press)*; Evidence for mechanical shear degradation of high polymers, *A. B. Bestul, J. Phys. Chem.* **61**, *418 (1957)*; Kinetics of capillary shear degradation in concentrated polymer solutions, *A. B. Bestul, J. Chem. Phys.* **24**, *1196 (1956)*; Composition of apparent shearing forces during shear degradation of polymers, *A. B. Bestul, J. Appl. Phys.* **25**, *1069 (1954)*.

[2] A worker consistometer for lubricating greases, *S. A. McKee and H. S. White, ASTM Bul.* **153**, *90 (1948)*. *See also, NBS Tech. News Bul.* **33**, *10 (1949)*.

[3] Effects of capillary shape on flow characteristics and degradation of polymer solutions, *H. S. White and H. V. Belcher, J. Research NBS* **60**, *215 (1958) RP2839.*

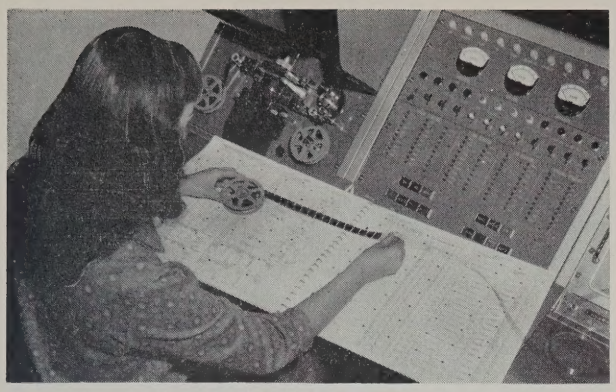


to assist in 1960 census

THE BUREAU has developed a new model of FOSDIC (Film Optical Sensing Device for Input to Computers) to assist the Bureau of the Census in processing the data to be received from the 1960 decennial censuses. This electronic machine rapidly reads microfilmed census documents and transcribes the data on magnetic tape for direct input to an electronic computer. It was designed by M. L. Greenough and C. C. Gordon of the NBS staff and E. S. Stein of the Bureau of the Census.

In the 1960 census, nearly 160,000 enumerators will be employed in collecting data on the people of the United States and their homes. The result of their work will be a vast amount of information about persons and households, amassed on nearly 50 million separate sheets of paper. To sum up all this information into convenient, useful tables and to produce this summation in the space of a few short months will require the services of four high-speed electronic computers.

Before the data is given to the computers for processing, an intermediate step must be performed. Each document must be read and the information on it translated into a language that the computers can understand. In the past, human operators have performed this task by transcribing the data onto punched cards. However, it was readily apparent that, for the 1960 censuses, the extremely rapid processing speeds which electronic computers can achieve would be largely wasted in the absence of some means for mechanizing the input. Therefore, the Bureau was called upon to develop an instrument that would eliminate as much as possible the human drudgery heretofore required. The result has been FOSDIC III. This machine is already in service, and has assisted in several census surveys. For the 1960 census, four additional iden-



Control console of FOSDIC III. The machine reads microfilmed census documents and transcribes the information onto magnetic tape for input for a computer. On the console are some of the original documents. Operator is holding some of the microfilmed copies. (Photo courtesy U.S. Bureau of the Census.)

tical machines are being constructed by the Bureau of the Census.

The central element of FOSDIC III, like its predecessors, FOSDIC I and II,[1] is an electronic scanning assembly. Light from the screen of a cathode-ray tube is focused upon the microfilm image, and the transmissivity of small, discrete areas on the film—corresponding to the handwritten marks on the original document—is measured with a photoelectric cell. By moving the electron beam around on the face of the cathode-ray tube, any selected area of the image can be examined. Control of the position of the illuminated area, and interpretation of the signals from the photoelectric cells, are functions performed by the associated electronic circuitry. Through positional control, or scanning, the point of light travels in prescribed manner from one point to the next in turn. Since there is only one electron beam in the cathode-ray tube, the scanning over the image is serial in its time sequence. Then, as soon as one image is scanned, the film is automatically advanced to the next frame.

Photographing the census documents on 16-mm microfilm provides a number of advantages. It separates the massive paperwork operation from the electronic equipment; FOSDIC can read microfilm images much faster than the paper sheets themselves. The high contrast of microfilm provides an enhanced signal-to-noise ratio, resulting in a low incidence of errors in machine reading. Moreover, film images of quality adequate for FOSDIC to read are also of sufficient quality for easy visual examination in a microfilm reader. Therefore, the original documents, once microfilmed, need not be saved; the storage problem is thereby reduced considerably. Documents can be photographed on a pile permitting rapid peeloff, the documents may be slightly tilted so that straightening is not continually required, and precise reduction ratio and exposure control are not necessary.

A system of notation easily recognizable by both man and machine is required for indicating information on census documents. The simplest system is "mark sensing", in which answer areas are marked with some writing instrument or left blank. The most familiar example is an "x" in a box to indicate a "yes", "no", or the correct answer to a multiple choice question. Numbers may be selected by choosing the desired digit from a column of 10 digits. Mark sensing permits the use of ordinary pencils, allows erasures, and minimizes the weight of paper stock which the census enumerator must carry with him.

A complete census document is built up of groups of questions, with their possible answers and answer areas. Each group is located by an index mark—simply a black square which FOSDIC can easily find. Answer marking positions fall into an imaginary grid laid off from the index. The grid is made up of modules 0.15 in. square, and marking areas may be packed this closely together, horizontally or vertically. The actual marking area is a fine-lined circle, one-half module in diameter. Each index serves a grid as large as 16 by 16 modules.

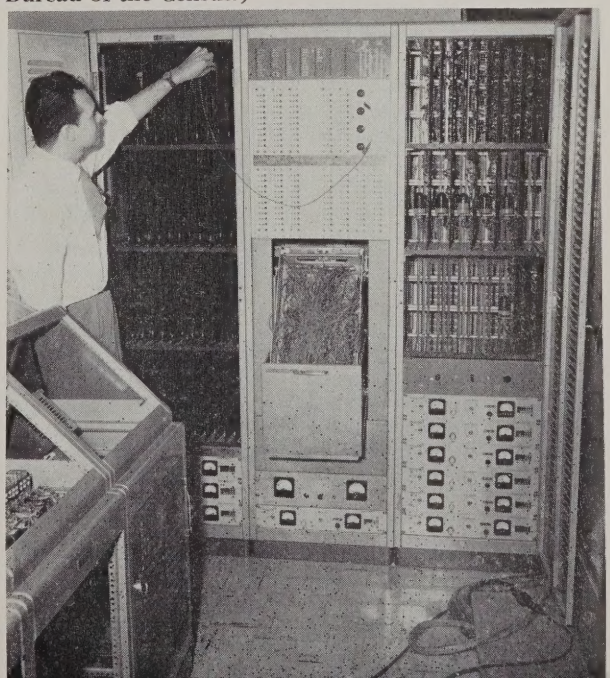
A document may contain, within its overall limitations of size, as many index groups as are needed for the information content. Each group is an entity in itself, and need have no relation in position with any other group.

Registration marks are located at the left and top borders of the document. These marks are included to determine the tilt and span of the document image as it appears to FOSDIC's scanning equipment, and to provide a starting point for examining the image.

FOSDIC can read off any part or all of the information on a document, depending on what the programer instructs the machine to do. The instructions are in terms of the horizontal and vertical coordinates of the index mark next to the desired information. Program control is through a plugboard into which the detection logic has been wired.

[1] FOSDIC—a film optical sensing device for input to computers, *NBS Tech. News Bul.* **38,** *24 (1954)*; FOSDIC II reads microfilmed punched cards, *ibid.* **41,** *72 (1957)*.

Racks containing part of FOSDIC's electronic equipment. The machine can read off part or all of the information on a microfilmed census document. Central element of the machine is an electronic scanning system that reads any selected portion of the film. (Photo courtesy U.S. Bureau of the Census.)

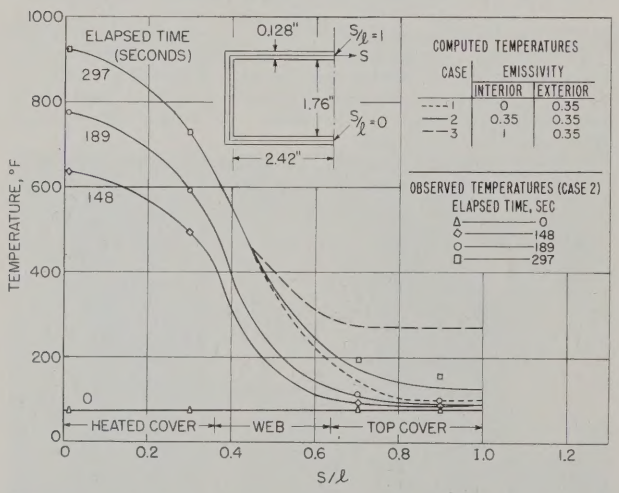


THERMAL STRESSES IN STAINLESS STEEL BOX BEAMS

INTERNAL radiant heat transfer in Type 302 stainless steel box beams has been investigated at the Bureau in work supported by the Office of Naval Research. The study was made to determine the extent to which this mechanism, as distinguished from external heat radiation, can alleviate the thermal stresses and deflections that can cause failure in supersonic aircraft and other structures subjected to elevated temperatures. The data show that significant relief may be obtained through maximum internal radiant transfer, a condition that can be achieved by blackening the internal surfaces of the structure. Designing aircraft beam structures to accomplish this effect should lead to savings in weight.

Thermal stress is basically dependent on temperature distribution. The steeper the thermal gradient is, the greater the thermal stress. This means that, for a fairly large difference in temperature between the heated and unheated beam covers, appreciable thermal stresses occur. Internal radiation, however, tends to lower the temperature gradient, and thus relieve the thermal stresses. Increasing the emittance of the internal surface of the heated cover tends to enhance this effect by increasing the rate of radiant heat transfer.

In the present study, the internal radiant heat transfer of 13 theoretical box beams at high-altitude conditions was computed by S. Goodman, S. B. Russell, and C. E. Noble.[1] These box beams were of three basic types, each varying in wall thickness. Variation with temperature of the thermal and elastic properties of the beam material was considered in the computations.

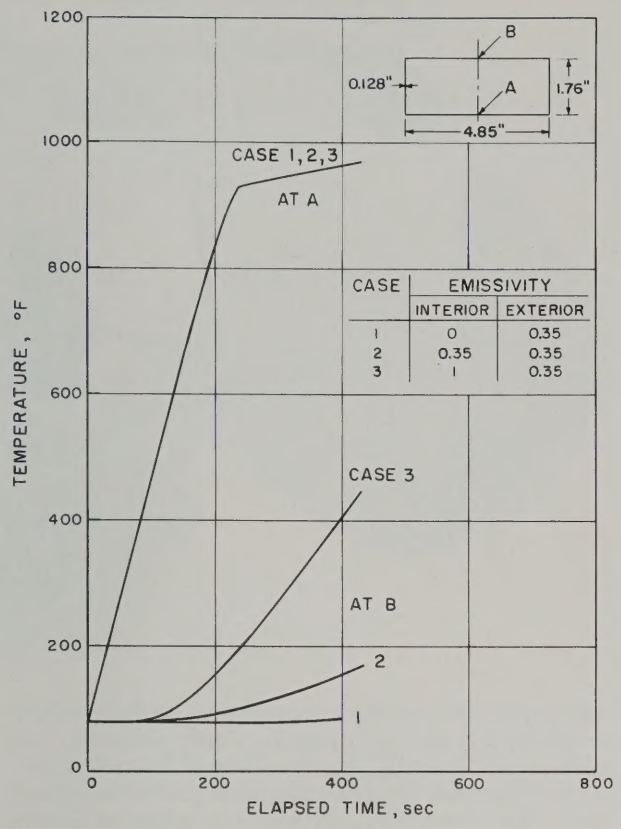


Computed and observed temperature distributions are compared in studying the effects of internal radiant heat transfer in stainless steel box beams. (S is a distance coordinate measured along the total length *l*, of the three sides of the left half of box beams as indicated in the diagram.)

A stainless steel box beam (center), used in studying internal radiant heat transfer, is being wired with several thermocouples to measure temperature distribution. The vacuum chamber eliminates convective heat transfer, simulating conditions at high altitudes.

Hemispherical total emittance was taken as 0, 0.35, and 1 for the internal surface and as 0.35 for the external surface—the zero value indicating minimum radiant heat transfer. Changes in the internal radiation were related to temperature distribution, maximum thermal stress, and beam deflection. The temperature distribution histories were computed on the Bureau's SEAC.

To check the theoretical data, one case was verified experimentally. The test procedure consisted in heating only one cover of a box beam in a vacuum chamber whose pressure was maintained at 4 mm of Hg. Vacuum conditions were used to minimize gas heat transfer. The beam was heated with quartz-tube tungsten filament heaters, which were enclosed in a silver-plated stainless steel reflector. The temperatures, measured at various intervals on the beam cover, were about 12 percent less at the beam ends than at the center of the cross section. Beam deflection was measured at the center of the cross section by a deflected cantilever carrying electrical strain gages.



Beam temperature history shows that maximum internal radiant heat transfer (case 3) significantly reduces the thermal gradient between the heated (A) and unheated (B) surfaces. The steeper the thermal gradient is, the greater the thermal stress.

Good agreement was obtained between the theoretical and experimental values for this beam. In evaluating the effect of a change in emittance on the thermal stress distribution and deflection, the possible effects of yielding, creep, and buckling were neglected.

The analysis showed that, for a thick-walled, slowly heated beam with a heated cover at 958° F, a change in the interior wall emittance has little effect on the magnitude of the maximum thermal stress. Increasing the interior wall emittance from 0 to 1.0, however, decreases the maximum beam deflection by 34 percent. For other beams at 1,200° F, the same change in emittance reduced the absolute maximum stress from 9 to 27 percent and deflection from 15 to 40 percent.

The study showed that maximum internal radiant heat transfer, indicated by an emittance value of 1, has an appreciable effect on the temperature distribution, and consequently on the maximum thermal stress and deflection. This effect is particularly marked for a thin-walled slowly heated beam.

[1] *For further technical details, see* Effect of internal radiant heat transfer on temperature distribution, thermal stress, and deflection in box beams, *by S. Goodman, S. B. Russell, and C. E. Noble, J. Research NBS* **62**, *No. 6 (1959) RP2949.*

NEW MOTION PICTURE

Dental X-ray Equipment: Alteration for Modern Radiation Hygiene

THE NEW motion picture was announced by the American Dental Association and the National Bureau of Standards, who have collaborated for 30 years in research on dental materials. The film is the ninth motion picture on dental materials produced by the Association's Council on Dental Research in cooperation with the Bureau. Available for loan or sale, the film, a 16-mm, sound, color film, running time 18 minutes, is designed for the dental and other health professions.

It is the first of a new series to be produced on radiation hygiene. The film shows the dentist how good diagnostic X-ray pictures may be produced with a minimum amount of radiation to the patient. Procedures by which the dentist can check his X-ray equipment for collimation and filtration of the useful beam, for tube head leakage, and for adequate length of the timer cord, are shown. It demonstrates how the dentist can use high-speed film with the some 80,000 X-ray machines currently available in dental offices. As illustrated in the new motion picture, ultrafast dental radiographic film may be used with a slow mechanical timer by reducing the milliamperage or by increasing the target-film distance. The recommendations are based upon those outlined in current dental literature and in the Bureau's radiation handbooks, prepared by the National Committee on Radiation Protection.

Arrangements for loan may be made by writing the Office of Technical Information, National Bureau of Standards, Washington 25, D.C., or the American Dental Association Film Library, 222 East Superior Street, Chicago 11, Ill.

Arrangements for purchase may be made by writing the Office of Technical Information, National Bureau of Standards, Washington 25, D.C. The current purchase price is $60.89 f.o.b. and is subject to change without notice.

503954—59——2

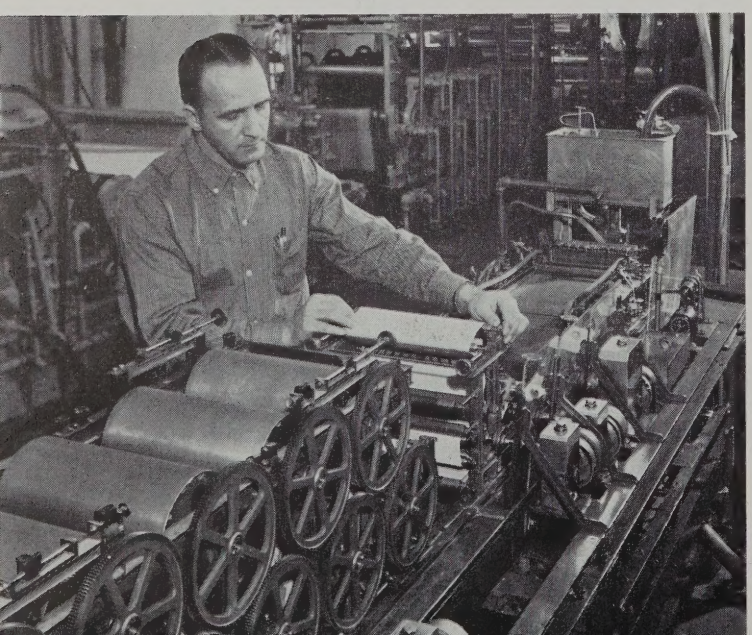
THIS YEAR marks the 50th anniversary of paper research at the National Bureau of Standards. In 1909 the Bureau first set up a laboratory to determine the physical and chemical properties of paper. Over the past half century a great many instruments and test methods have been developed to measure these properties, providing a firm basis for purchase specifications that are used today for paper and paper products.

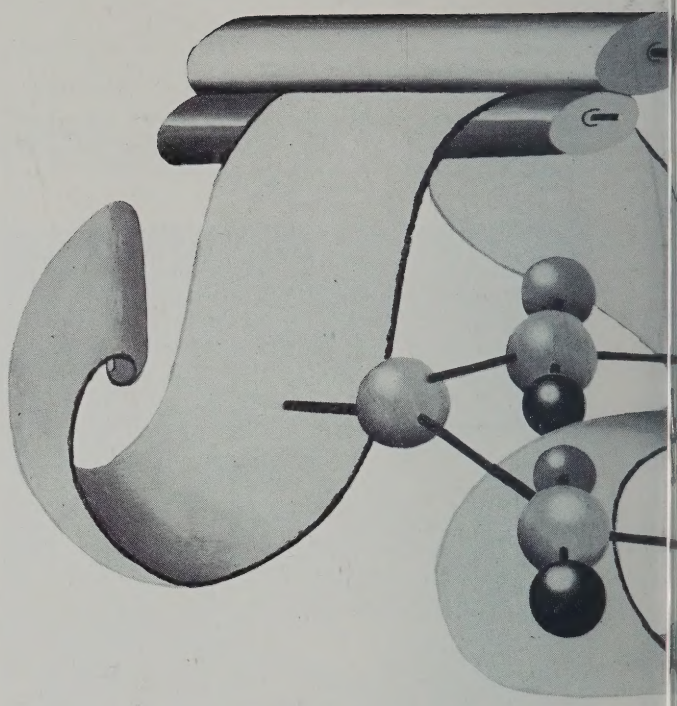
When the Bureau began this program the paper industry was relatively undeveloped. But today the pulp and paper industry is one of the largest in the country. More than 30 million tons valued at $10 billion are produced annually. Per capita consumption exceeds 400 lbs a year. The government alone buys more than 200 kinds of paper annually for its own purposes at an estimated cost of $100 million.

As the paper industry has developed, the character of the Bureau's program has changed. Less emphasis is now placed on developing test methods and paper processes. Current paper research, under the direction of R. B. Hobbs, seeks to provide basic information on the properties of cellulose and other polymeric substances from which papers are made. Such information is used to improve the mechanical, optical, and performance qualities of paper. Studies are also being conducted to develop standard reference materials, with known physical properties such as tearing strength or folding endurance, which can be used in calibrating commercial testing instruments. Thus, the paper research program is continuing to play a significant role in the advancement of paper technology.

Physical Properties

To obtain the best results in service, the physical properties of paper must be closely controlled. In printing papers, for example, brightness (a measure of apparent whiteness), opacity (related to "see-through"), and gloss are necessary optical properties. Good folding endurance and bursting and tearing strengths are needed in many types of paper designed to give service for an extended period. Food-packaging papers and building papers must be impermeable to water, air, and water vapor. Ready absorption and softness are essential characteristics of facial tissues.





An important attribute of papers processed through packaging machinery, or used for magazine covers, paper boxes, and the like, is stiffness. A method and apparatus were developed for measuring stiffness,[1] and the equipment is now available commercially. In this device, a specimen is bent through a given angle, and its stiffness is measured as the torque in a wire suspension. By varying the size of the supporting wires and the angle through which the specimen is bent, papers having a wide range of stiffness can be tested.

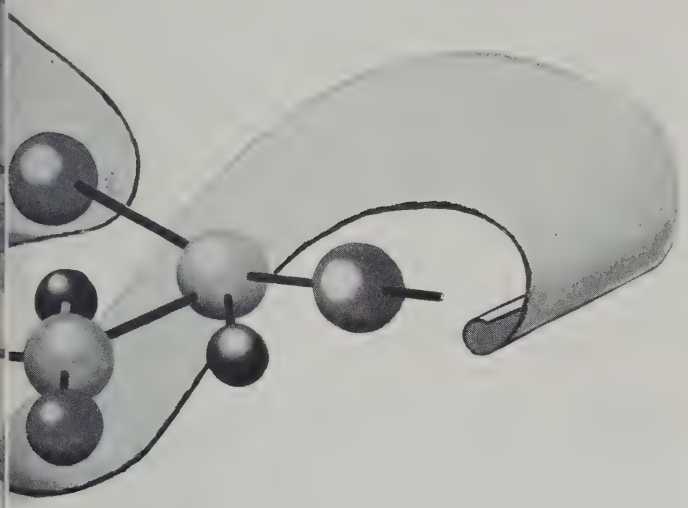
Smoothness is a factor used to determine the printing quality of paper. In a comparative study of two paper smoothness testers, the first practical application was made of the "sensitivity criterion".[2] With this concept the relative merit of the two instruments is compared by the ratio of their sensitivities. The criterion is especially useful when no absolute standard of comparison is available and no simple proportional relationship exists between the results obtained with two different instruments.

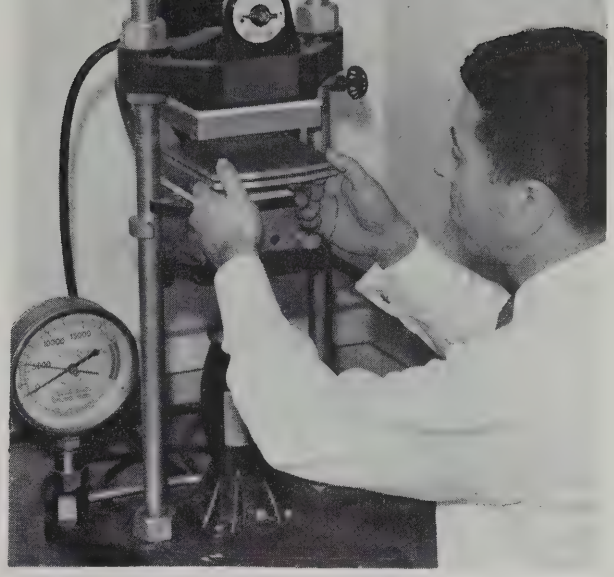
Some government departments have unique paper requirements. Perhaps the most obvious example is the paper used for making U.S. currency. Other examples are high wet-strength map papers and postage stamp papers. A new process of dry intaglio printing recently installed at the U.S. Bureau of Engraving and Printing required a paper soft enough to reproduce the fine lines of the engraving plates yet durable enough to last at least as long as currency printed by a wet intaglio process. NBS devised laboratory tests to

A miniature Fourdrinier papermaking machine is used at the Bureau for experimental production of small quantities of paper. Adjustments are being made to the dryer felt that carries the paper to the dryer section.

NBS Technical News Bulletin

PAPER RESEARCH





In experiments to determine the effectiveness of lamination with acetate to preserve historical documents, a flat press is used. Here a metal "sandwich" holding the document and the film is inserted into the press.

evaluate experimental papers submitted by the industry, and the results were used to select a paper for wide-scale production. A statistical study is now being made to compare the durability of the new and old papers in actual use.

Map papers for the military services must be exceptionally resistant to hard wear because of the excessive folding, crumpling, soiling, and extreme weather conditions to which the maps are exposed. During World War II, the Bureau worked with the Army and paper manufacturers to develop a paper with high wet strength and other durable properties. By incorporating synthetic resins in the paper mixture, the desired result was achieved.

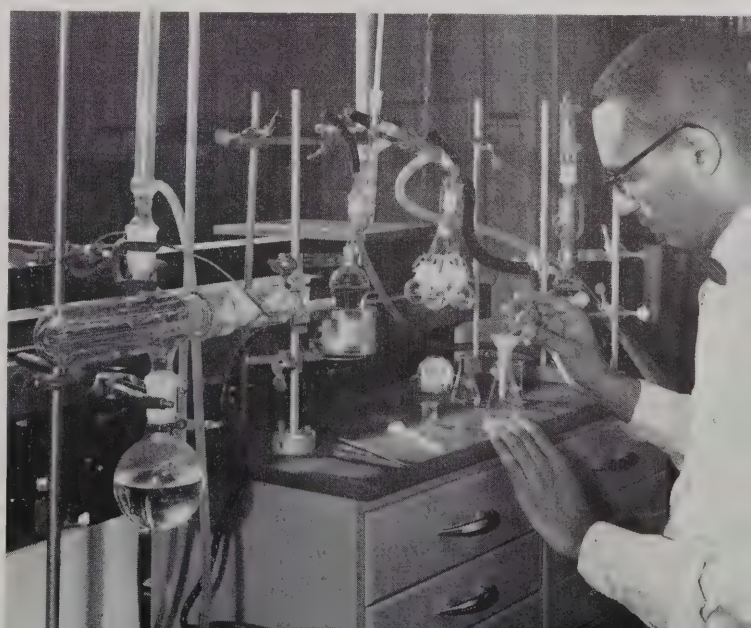
One of the first studies on the physical properties of paper was made by D. E. Douty of the paper laboratory. His report on the bursting strength of paper was published in 1910. Since then a large number of Bureau reports have been published on the physical properties of paper and standard methods for measuring these properties.

World War I brought about activity in the preparation of government specifications, and the need for new paper specifications increased considerably in the 1920's. During this period the late B. W. Scribner, then chief of the paper laboratory, was appointed chairman of the interdepartmental committee for Federal specifications. Pioneer work was accomplished in preparing specifications for more than a hundred different kinds of paper. After World War II, however, administrative responsibility for this function was transferred to the General Services Administration.

In the period from 1920 to 1935 considerable effort was spent in investigating the materials and processes used in industrial papermaking. The program was undertaken to assist in developing a strong American industry by reducing the Nation's dependence on sources of supply outside of North America. The requirement for this work diminished as the industry grew and prospered, and as university and commercial laboratories took over the necessary investigations.

Chemical Properties

The utility and value of paper for many of the ways in which it is employed depend on its chemical stability and, consequently, on the stability of cellulose, its major constituent. For example, U.S. Supreme Court Reports are printed on a paper consisting of 100-percent new cotton fibers. The paper is processed with special care to minimize residual chemicals so that the Court's decisions will be available for centuries in permanently bound form. A knowledge of the chemical structure and reactions of cellulose is basic to the manufacture of paper requiring such a high degree of durability, as well as to the preservation and storage of historical records.



Apparatus used to study organic reactions that occur in the resin-bonding of fibers.

Handsheet apparatus used for preparation of experimental papers. The operator raises the cylinder, containing a measured amount of aqueous pulp suspension, to remove the formed sheet.

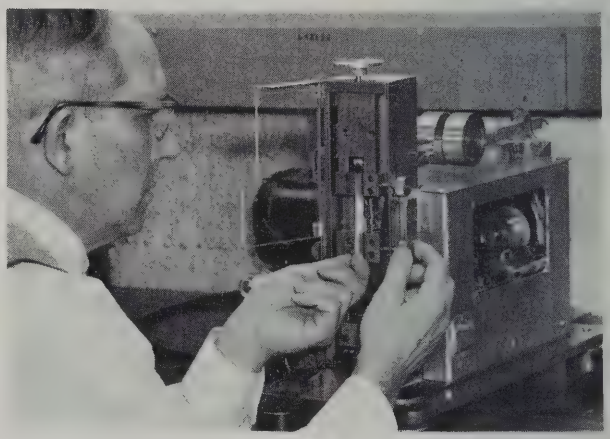
For several years the Bureau has investigated the chemistry of cellulose, particularly its degradation by environmental conditions. Studies have been made of oxidation with periodic acid;[3] reaction with sodium chlorite;[4] thermal degradation;[5] and degradation by ultraviolet light.[6] Among the analytical methods that have been investigated are those for copper number;[7] alpha, beta, and gamma cellulose;[8] *p*H;[9] and functional groups in cellulose.[10]

Recently, research has been conducted for the Army Engineer Research and Development Laboratories to reduce the hygroscopic expansivity of map paper,[11] and thus improve its dimensional stability for reproducing several colors in successive printings. The two most promising processes found—lamination with a polyester film, and use of synthetic organic fibers in the paper blend—are too costly for immediate practical use. However, it is anticipated that fundamental research now in progress on the nature of resin-to-cellulose bonding will lead to a satisfactory method for dimensional stabilization.

In 1951 the Bureau developed techniques for making paper entirely from glass fibers in cooperation with the Naval Research Laboratory.[12] This paper is now widely used, and millions of dollars are invested annually in its production on a commercial scale. It serves as a filter for removing solid particles from gases, and hence is used principally in gas masks and other atmospheric filters. Glass-fiber paper is also employed both as a chemical filter and a dielectric material.

The preservation and restoration of documents are of considerable interest to the National Archives, the Library of Congress, state and municipal records offices, and insurance companies. The Bureau has worked with these groups on problems such as the effect of the chemical content of paper on its aging; the care of books and films in libraries; the preservation of the United States Constitution and Declaration of Independence; and the lamination of documents with cellulose acetate film. Methods for studying the aging of acetate film have been developed,[13] and improved specifications for laminating film have been prepared.

A study of papers made from groundwood fibers showed that a clay coating, which is sometimes applied to give a better printing surface, affords considerable protection against light.[14] These fibers, used in newsprint and other papers with a short life expectancy, are one of the least expensive papermaking materials. Their use has been limited, however, because they turn yellow when exposed to light. It was found that the application of commercial coatings, made of finely divided clay, mixed with either starch, glue, casein, or synthetic resin as a binder, reduces the deteriorating effects of light by about 50 percent. These results have helped to increase the use of groundwood fibers in papers requiring a moderate degree of permanency.



This apparatus for studying the folding endurance of paper was recently developed at the Bureau. A small paper specimen is being inserted into the machine.

Test Methods and Instruments

Two problems of present concern to the paper industry are: (1) How to determine which of two test methods gives the more sensitive and precise results; and (2) how to explain the lack of agreement in the results obtained by different laboratories using the same test method on the same paper. In cooperation with the Technical Association of the Pulp and Paper Industry (TAPPI) and the American Society for Testing Materials, the Bureau is engaged in comparative studies of different methods, utilizing the sensitivity criterion as a statistical tool. Collaborative work on chemical test methods is being conducted with the American Chemical Society and the International Committee for Cellulose Analysis. In an effort to achieve better agreement between laboratories, a mathematical theory underlying the comparison of results from different laboratories is being applied to interlaboratory comparisons of test methods.

In cooperation with TAPPI and the American Paper and Pulp Association, studies are in progress to develop a standard sample of paper that may be used for calibrating tearing strength testers. If this standard succeeds in reducing interlaboratory inconsistencies, additional standards will be established for calibrating other test instruments.

A higher degree of refinement is always being sought for almost all types of standards, and this is true of paper standards. A few years ago papermaking machines operated at speeds of a few hundred feet per minute. Today speeds of over 2,000 ft/min are easily attained, and printing and converting processes have undergone similar acceleration. Hence, more rigorous specifications are required for the physical properties of paper, as well as new test methods for measuring dynamic rather than static properties. New varieties of paper developed from inorganic and synthetic fibers, and from new nonfibrous materials, also impose demands on all technical organizations connected with the paper industry. The fundamental information now being developed in the Bureau's research effort should prove useful in devising techniques to solve the problems presented by these new trends.

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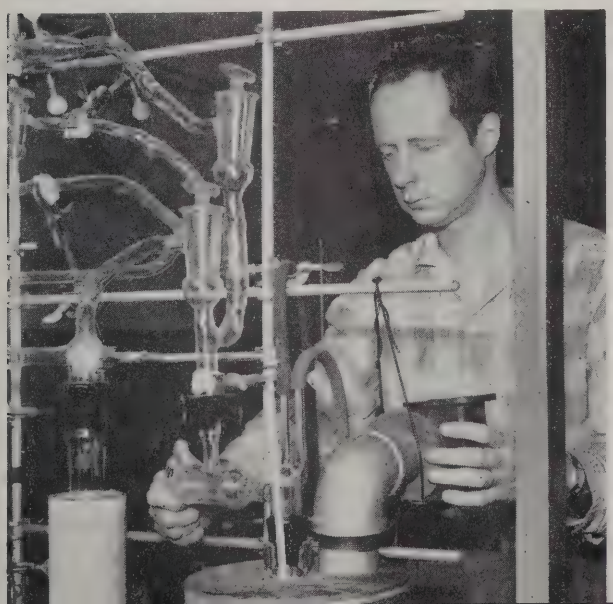
[1] The stiffness of paper, *by F. T. Carson and V. Worthington, TAPPI* **36,** *36 (1953)* ; Stiffness tester, *NBS Tech. News Bul.* **36,** *182 (1952)* ; Extending the range of the NBS stiffness tester, *by V. Worthington, TAPPI* **37,** *384 (1954)*.

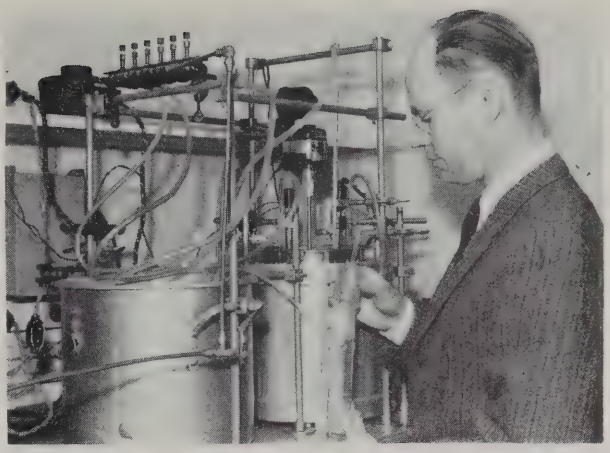
[2] Use of the sensitivity criterion for the comparison of the Bekk and Sheffield smoothness testers, *by T. W. Lashof, J. Mandel, and V. Worthington, ibid.* **39,** *532 (1956)* ; Evaluation of test methods by the sensitivity criterion, *NBS Tech. News Bul.* **40,** *139 (1956)*.

[3] Reaction of sodium chlorite with some aldoses and modified celluloses, *by W. K. Wilson and A. A. Padgett, TAPPI* **38,** *292 (1955)*.

[4] Determination of glucose by means of sodium chlorite, *by H. F. Launer, W. K. Wilson, and J. H. Flynn, J. Research NBS,* **51,** *237 (1953) RP2456;* Determination of aldehyde in glucose, *NBS Tech. News Bul.* **38,** *22 (1954)*.

Investigating the degradation of cellulose by ultraviolet light. Adjustments are being made to the stopcock that connects the reaction vessel and irradiation apparatus to a vacuum manifold.





Apparatus used to accelerate the aging of plastic specimens. An aging vessel containing cellulose acetate specimens is assembled before immersion in the oil bath on the right. On the left is a water bath containing bubblers for humidification of oxygen before it is passed through the aging vessel.

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[5] Thermal degradation of cellulosic materials, *by S. L. Madorsky, V. E. Hart, and S. Straus, J. Research NBS* **60,** *343 (1958) RP2853.*

[6] Degradation of cellulose in a vacuum with ultraviolet light, *by J. H. Flynn, W. K. Wilson, and W. L. Morrow, ibid.* **60,** *3 (1958) RP2841;* Degradation of cellulose by ultraviolet light, *NBS Tech. News Bul.* **42,** *169 (1958)*.

[7] A modified method for the determination of copper number in paper, *by B. W. Scribner and W. R. Brode, Tech. Pap. BS* **22,** *9 (1927–28) T354;* The determination of alpha-cellulose content and copper number of paper, *by J. O. Burton and R. H. Rasch, BS J. Research* **6,** *603 (1931) RP973.*

[8] Simplified volumetric determination of alpha, beta, and gamma cellulose in pulps and papers, *by H. F. Launer, J. Research NBS* **18,** *333 (1937) RP979;* Volumetric determination of alpha, beta, and gamma cellulose in pulps and papers containing sizing, filler, and other materials, *by H. F. Launer, J. Research NBS* **20,** *87 (1938) RP1068.*

[9] Determination of the *p*H value of papers, *by H. F. Launer and W. K. Wilson, J. Research NBS* **23,** *663 (1939) RP1262.*

[10] Aldehyde, carboxyl, pentosan, and degree of polymerization data on the ICCA standard pulps, *by W. L. Morrow, TAPPI (in press)*.

[11] Properties of some experimental map papers containing synthetic fibers *by G. L. McLeod, ibid,* **41,** *430 (1958)* ; *and NBS Tech. News Bul.* **43,** *8 (1959)* ; Improved dimensional stability in laminated map paper, *by G. L. McLeod and T. L. Worksman, TAPPI* **41,** *484 (1958)* ; *and NBS Tech. News Bul.* **42,** *148 (1958)*.

[12] The electrical properties of glass-fiber paper, *by T. D. Callinan, R. T. Lucas, and R. C. Bowers, Elec. Mfg.* **48,** *94 (1951)* ; Paper from glass fibers, *NBS Tech. News Bul.* **35,** *177 (1951)* ; Effects of mechanical preparation and *p*H on the strength of glass-fiber paper, *by M. J. O'Leary, R. B. Hobbs, J. K. Missimer, and J. J. Erving, TAPPI* **37,** *446 (1954)* ; Improved glass-fiber paper, *NBS Tech. News Bul.* **39,** *82 (1955)*.

[13] Degradation of cellulose acetate films, *by W. K. Wilson and B. W. Forshee, SPE Journal (in press)* ; Preserving documents by lamination, *NBS Tech. News Bul.* **42,** *54 (1958)*.

[14] Effect of light on coated groundwood fibers, *by W. K. Wilson and J. L. Harvey, TAPPI* **36,** *459 (1953)* ; *NBS Tech. News Bul.* **38,** *155 (1954)*.

Symposium on the Optical Pyrometer Temperature Scale

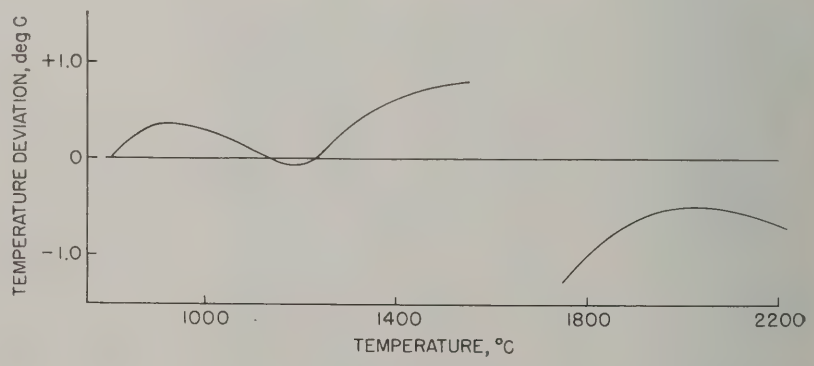
TO FACILITATE the exchange of information and ideas on high-temperature pyrometry, a Symposium on the Optical Pyrometer Temperature Scale was held at the Bureau, October 23 to 25, 1958. Sponsored by the Heat Division of the Bureau and the High Temperature Group of Argonne National Laboratory, this Symposium concentrated on present temperature scales and calibration practices, recent improvements in optical pyrometer techniques and instruments, and the possible use of the temperature scale beyond 4,000° K. Chairman of the Symposium was C. M. Herzfeld, Chief of the Heat Division.

In the last few years, the interest in high-temperature measurements has created a demand for optical pyrometers capable of greater precision and accuracy. This need has raised many questions about procedures for realizing the International Temperature Scale at high tempertures (above 1,063° C) and about the calibration, use, and manufacture of optical pyrometers. Recent studies, principally at the National Bureau of Standards and Argonne National Laboratory, have shed new light on some of these problems. To communicate these ideas as rapidly and conveniently as possible, and at the same time provide an opportunity for discussing individual problems, the Symposium on the Optical Pyrometer Temperature Scale was planned. That the need for such a program had long been felt was evidenced by the nationwide response from the industrial and research laboratories performing significant work in this field.

Trends in the Bureau's work on optical pyrometry were outlined at the beginning of the conference by Dr. Herzfeld. He pointed out the necessity of improving methods of temperature measurement in the range 800° to 4,000° C under ideal conditions. Here accuracy, precision, and convenience of operation need further improvement. An extension of methods for measuring temperatures under ideal or approximately ideal conditions in the range 4,000° to 10,000° C is also required. In addition, improvements in temperature measurement under nonideal conditions, such as in flames and complicated systems, are needed along with more fundamental research leading to new and better ways of measuring temperature.

To form a basis for further discussion, a summary of the theory of optical pyrometry was presented by H. J. Kostkowski of the Bureau and G. H. Winslow of the Argonne National Laboratory. The various assumptions concerning pyrometer calibration were carefully analyzed. The importance of the relative visibility function of the observer and the adequacy of using a tungsten strip lamp rather than a blackbody for temperatures above the gold point were emphasized.

This paper was complemented by a discussion of the calibration of the National Bureau of Standards standard pyrometer, presented by Bureau staff members H. J. Kostkowski and R. D. Lee. A recent attempt to realize the International Temperature Scale with maximum precision by a careful *primary* calibration was described. The standard optical pyrometer, a disappearing filament type, is calibrated at the gold point by sighting on a blackbody maintained at the freezing point of gold, one of the primary fixed points on the International Temperature Scale. At higher or lower temperatures the calibration is achieved by sighting at a tungsten strip lamp through rotating sector disks and using the Planck radiation law to calculate the unknown temperature. This calculation also involves the mean effective wavelength, which is determined by the filters in the optical pyrometer and the relative visibility function of the observer. Greater reliability was achieved in this calibration by determining independently the visibility function of each observer rather than using an average visibility curve as is done in most standards laboratories. It is felt that the results of this primary calibration are about the best one can expect from visual optical pyrometry, with uncertainties in the calibration going from ± 0.5 deg C at the gold point to ± 4 deg C at 2,400° C. An international comparison of strip lamps reveals that this optical pyrometer scale agrees with the mean scale of the national laboratories of Canada, West Germany, and East Ger-



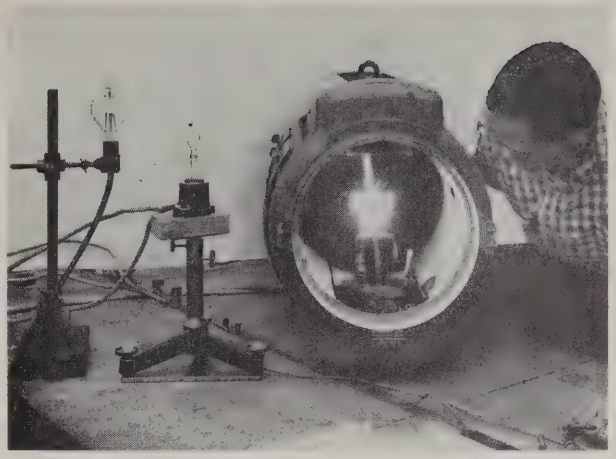
Results of the 1958 international comparison of strip lamps by the National Bureau of Standards and the national standards laboratories of Canada, West Germany, and East Germany. The difference between the Bureau's calibration and the mean of the other laboratories is 1.3 deg C or less.

many to within ±1 deg C in the range compared (800° to 2,200° C).[1]

The procedure employed at Argonne to establish the temperature scale in the range of 1,000° to 2,500° C was discussed by R. J. Thorn and G. H. Winslow of that laboratory. The starting point for the calibration was the freezing point of copper, which has been determined as 1,083° C on the 1948 International Temperature Scale. Above the freezing point of copper a blackbody constructed of tungsten, contained in a vacuum and heated inductively, was employed as a light source. Some advantages of using a blackbody rather than a tungsten strip lamp were discussed. Effective wavelengths, calculated from the measured transmissions of the pyrometer red filter and the standard visibility function, were presented. The Bureau and Argonne National Laboratory optical pyrometer temperature scales agree within 1° C at the gold point, but differ by 4° C above 1,600° C. It is suggested that the most likely source of the difference lies in the effective wavelengths.

The basic limitations on the accuracy of optical pyrometry were discussed by D. R. Lovejoy of the National Research Council of Canada. An estimate of the errors arising in primary standard pyrometer calibrations and in secondary standard strip lamp calibrations was shown to agree with the results of a recent intercomparison of secondary strip lamps. An evaluation of a carbon arc, accepted as a useful secondary standard at 3,514° C, showed that in this region the effect of the uncertainty in effective wavelength is greater than all other sources of error combined. Even though very precise work was involved in determining the platinum point at 1,769° C, metal freezing points seem to offer little advantage over the strip lamp as secondary standards. This paper concluded with a discussion of the errors in calibrating nonstandard pyrometers, showing that good commercial pyrometers can be calibrated with a precision comparable to that of primary standard pyrometers if the lamp current is calibrated directly rather than through the internal potentiometer and scale.

R. D. Lee of the Bureau discussed the procedures used in calibrating optical pyrometers and tungsten ribbon-filament lamps submitted to the Bureau. Temperature comparisons are made with the NBS standard pyrometer from 800° to 3,800° C. In calibrating optical pyrometers, a tungsten ribbon filament lamp is used as a temperature source up to a brightness temperature of about 2,400° C, and a zirconium arc and carbon arc to temperatures of 2,800° and 3,800° C, respectively. Since the brightness temperature of a nonblackbody is determined in part by the red filter glass of a pyrometer, corrections are made for test pyrometers with different transmission characteristics. The estimated uncertainty of the calibration below 2,800° C is based on experience with a number of instruments rather than on the specific data of a given instrument. The standard deviation, with respect to the International Temperature Scale, of pyrometer and strip lamp calibrations is about ±1 deg from 800° to 1,500° C, ±2 deg from 1,500° to 2,000° C and increases to about ±6 deg at 2,800° C.



Temperature sources used in the Bureau's calibrations of optical pyrometers. A tungsten ribbon-filament lamp (center) provides brightnesses corresponding to temperatures up to 2,400° C. Calibrations at higher temperature are made with a zirconium arc (left) and a carbon arc (right). These sources have brightnesses of 2,800° and 3,800° C, respectively.

Methods of precise temperature measurement in determining the properties of refractory materials at the Los Alamos Scientific Laboratory were described by M. Bowman. Measurements of rapidly changing temperatures and of small temperature changes are required for the technique employed in measuring thermal diffusivity. These temperature changes are obtained by means of a multiplier phototube and a high-speed recorder.

L. Brewer of the University of California concentrated on the adequacy of the temperature scale in the determination of thermodynamic properties. The adequacy depends on the point of view; that is, whether the second or third law of thermodynamics is used in the calculation of thermodynamic variables. Brewer suggested that the accuracy requirements for a temperature scale to be used with the second law are extremely rigorous. In a third law method, where the heat of formation and the temperature are proportional, an uncertainty in the absolute temperature of 0.5 percent is a reasonable goal. This paper also reviewed at some length past problems arising with NBS calibrations of certain optical pyrometers. Greater care in the use of these instruments and recent improvements in calibration practices seem to have eliminated these problems.

H. F. Stimson of the Bureau discussed the relation of the International Temperature Scale to the Thermodynamic Scale. This paper included a report on the present state of agreement between the two scales and work being carried out to achieve a better knowledge of the difference between these scales.

The present techniques employed in the design, manufacture, and calibration of commercial pyrometers

were outlined by K. A. Walch and R. C. Stroud of Leeds and Northrup, and by Paul V. Bollerman of the Pyrometer Instrument Company.

A brief summary of work going on at the Bureau on photoelectric pyrometers was given by C. P. Johnson and D. Erminy.[2] The first model of the NBS photoelectric pyrometer has a sensitivity of 0.1 deg C at the gold point and a spectral bandwidth of only 100 A. These features, together with its objective character, make it an attractive instrument. However, the measurement of brightness temperatures to a precision of 0.1 deg C introduces many difficult problems involving lens aberration, scattered light diffraction, and the stability of light sources. These must be investigated in great detail before the photoelectric pyrometer can be considered a reliable, accurate instrument.

Concluding the discussion of high-temperature measurements was a report on measurements of the color temperatures of a number of stars by E. Kron of the Lick Observatory, University of California. This work may have some future bearing on the calibration and measurement of very-high-temperature sources.

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[1] International comparison of high-temperature standards, *NBS Tech. News Bul.* **42,** *140 (1958).*

[2] High-temperature measurements, *NBS Tech. News Bul.* **42,** *125 (1958).*

Low-Temperature Distillation of Hydrogen Isotopes

EXPERIMENTAL work at the Bureau's Cryogenic Engineering Laboratory (CEL) at Boulder, Colorado, has advanced a practical solution to the problem of separating hydrogen isotopes.[1] A pilot plant capable of distilling liquid hydrogen by fractionation has yielded specific amounts of pure liquid deuterium and deuterium-free hydrogen.

The unit, the only one of its kind in the country, was designed by T. M. Flynn, K. D. Timmerhaus, and D. H. Weitzel. The designers feel that it is feasible for industry to produce large quantities of deuterium by this cryogenic distillation method; it is, in fact, particularly well suited for industrial areas where extensive supplies of hydrogen gas are available—near ammonia plants.

Of the three isotopes of hydrogen—protium, deuterium, and tritium—protium, the lightest, is the most abundant; over 99 percent of available hydrogen is in the form of protium. Tritium, extremely rare in nature, is radioactive and has a half life of about 12½ years. Deuterium, twice as heavy as "ordinary" hydrogen, occurs naturally in an abundance ratio of 1:7,000.

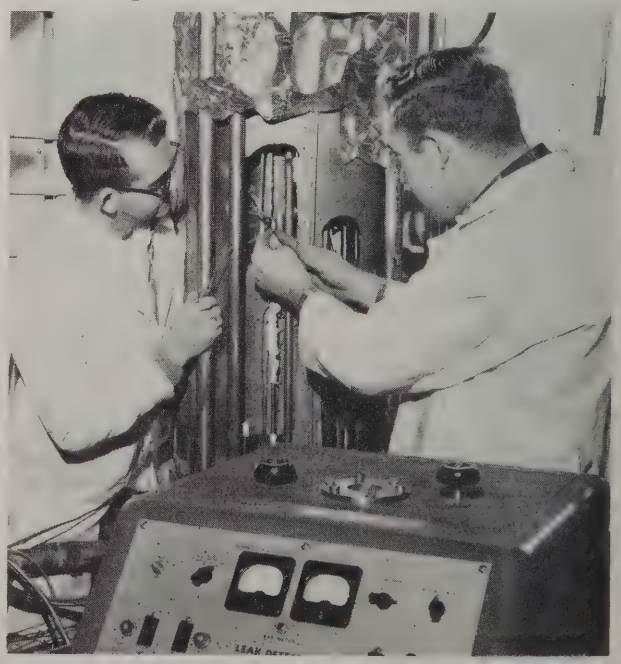
The demand for deuterium is growing. Recent declassification of thermonuclear fusion research programs has shown deuterium to be one of the most promising "fuels of the future". Furthermore, "heavy water", or deuterium oxide, has been one of the most desirable nuclear reactor moderators ever since the early days of the Manhattan Project. At that time, unavailability and economies restricted its use. At present, synthesis of heavy water is carried out on a commercial scale.

Deuterium-free hydrogen, although currently confined to research-quantity needs, is required in amounts sizable enough to necessitate pilot plant manufacture. For instance, bubble chambers, including those containing liquid hydrogen, are supplementing the Wilson Cloud Chamber and similar detection equipment used in elementary particle investigations. For this research some scientists demand deuterium-free liquid hydrogen; others use liquid hydrogen containing all isotopic modifications.

A few of the more common methods employed to manufacture heavy water or to separate hydrogen isotopes have certain disadvantages when put on a large scale. Consider the separation factor, the ratio of the vapor pressures of the two constituents at the boiling point of the system, as an index of merit. If the factor is high enough to give good separation, then the system involved is worthy of consideration. If, however, the factor is below the limits of practical consideration, the system is inefficient. The value for the heavy and "normal" water ratio is 1.026. Therefore, it is easy to realize why separation of heavy from "normal" water has drawbacks. One is that a great number of distillation stages are needed to produce a high concentration of HDO or D_2O (deuterium is present in water as HDO; as it becomes concentrated, it dissociates as follows: $2HDO \rightarrow H_2O + D_2O$). Secondly, heating and cooling requirements are very large.

If separation is to be by electrolysis, several hundred stages are necessary for production of pure heavy water.

Leak detection in a pressurized cryogenic system is extremely important. *Left:* Helium gas is being applied around the column in the search for "high vacuum" leaks. *Right:* Technician smooths a plastic sponge over an area suspected of minute leaks.



The dual-temperature process, used by a number of heavy-water manufacturers, depends upon an equilibrium between certain gases, usually hydrogen and hydrogen sulfide, to yield a concentration of deuterium higher in one than in the other. Hydrogen sulfide is corrosive and poisonous to both human beings and catalysts. The hazards connected with using hydrogen sulfide plus the length of time that would be involved if no catalyst were used (200 days minimum) make this catalytic exchange method unsuitable.

The separation factor for the hydrogen isotopes (2.26) suits ordinary distillation standards based on relative volatilities. However, until recently, large-scale distillation for purposes of hydrogen deuteride recovery had been rejected. The main deterrents responsible for this situation were lack of design data and the problems encountered in operating units at the extremely low temperatures that are required for distilling liquid hydrogen. In spite of this, CEL engineers felt that by using recent advances in cryogenic technology they could design an experimental program that would yield a practicable and efficient system. Paper studies made concurrently with early Manhattan Project investigations of the heavy-water situation substantiated their beliefs.

Preliminary experiments to collect data necessary in designing low-temperature separation columns started at CEL in 1955. Messrs. Flynn, Timmerhaus, Weitzel, and Vander Arend initiated the program to evaluate some of the parameters, such as plate efficiency and pressure drop. Their design of three small columns to operate at −423° F led to improved suggestions for a column or series of columns intended to increase the concentration of hydrogen deuteride in hydrogen from its natural abundance (1 part in 3,500) to any desired amount. Raising an HD:H concentration from the initial value to approximately 3 parts in 100 is the most difficult phase of the separation procedure. In designing their pilot plant to accomplish this specific job, CEL scientists assumed that parameters obtained experimentally for an H_2–D_2 system would be applicable to an H_2–HD system. In order to obtain the necessary values, they varied the number and types of plates for the column. One column was packed with stainless steel helices and the other two fitted with perforated plates.

To adapt the column to a limited space, the designers chose a high external reflux ratio. This ratio, a comparison between the volume of vapor returning to the column and the distillation product, is derived from the vapor-liquid equilibrium curve calculated for a particular column. Even though high-reflux conditions are difficult to achieve in a cryogenic system, the designers used a ratio value of 2.0—40 percent greater than the minimum calculated for their column. The result is a reduced column height. In addition, the experimental value of the column is not downgraded.

Likewise, for space reasons, the experimenters made the number of plates as small as possible. Use of the McCabe-Thiele theory, a conventional graphical method for determination of the appropriate number of plates for a particular distillation problem, provided the engineers with the exact number necessary.

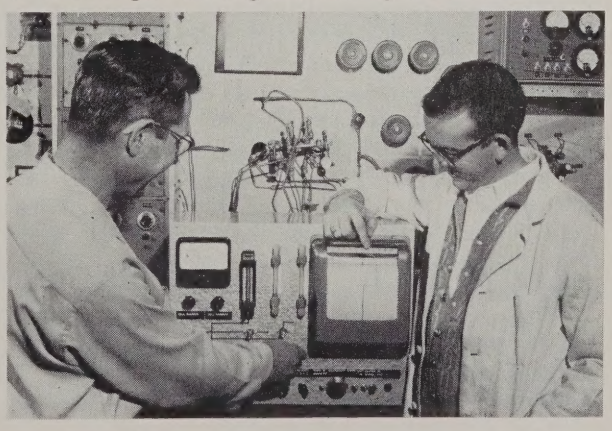
The difference in column diameter between laboratory-scale and pilot-plant-size equipment is somewhat arbitrary. However, an increase from 1½ to 6 in. places the column in the pilot-plant category. This pilot-plant size yields information directly applicable to commercial-size units. In terms of an equivalent heavy-water production rate, the feed rate of this pilot-plant column represents approximately 45 lb of heavy water for an 8,000-hr year.

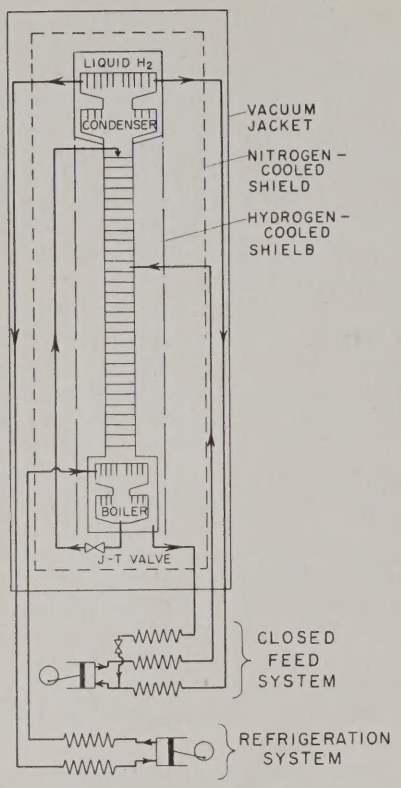
The flow system of the column consists of three separate parts—circulation, condenser, and feed. In the circulation system, a heat pump, operating between the condenser and the reboiler, provides the necessary refrigeration at the top and associated boil-up in the reboiler. Hydrogen gas, the circulating refrigerant, transfers heat from the condenser to boiler. This operation supplies sufficient heat to fulfill the requirements of the system.

The condenser and reboiler systems are similar; the former has a finned heat exchanger and a reservoir, or heat sink, of liquid hydrogen. This furnishes the other portion of hydrogen needed for refluxing. As the reflux condenses, the hydrogen vaporizes under atmospheric pressure. A resistance-type liquid-level sensing device and a solenoid valve on the transfer line keep the reservoir filled automatically.

Many of the problems encountered in designing this pilot plant are characteristic of standard distillation operations. However, a few arose which are peculiar to low-temperature units. For instance, in high-temperature units, heat loss is from the column. As a result, some additional vapor will condense to a liquid within the column. This means an increase in the internal reflux ratio. In addition, this transfer of energy (away from the column) requires that the size of the reboiler be increased to compensate for this heat loss. In low-temperature operations the effect is just the opposite; that is, a decrease in the internal reflux

To observe the liquid-hydrogen distillation system, a thermal conductivity gas analyzer was incorporated into the pilot plant. This extremely sensitive instrument reliably indicates the percentage of deuterium found in streams of gas entering and leaving the column.





Flow system of the pilot plant for low-temperature distillation of hydrogen isotopes.

and a corresponding decrease in the hydrogen-deuterium separation. Since stepping up the refrigeration efficiency of the system is expensive, CEL engineers designed a compact "cold box", crowded with components, to minimize heat leak. The column and accessories are suspended inside a vacuum space, from a stainless steel tripod supported on a bottom plate. This practically eliminates two energy transfer problems: (1) When the vacuum is of the order of 10^{-6}- to 10^{-7}-mm Hg, heat transfer by gaseous conduction is negligible; and (2) conduction through solids is small when supports are designed in the form of long, low-conductivity heat paths.

The pilot plant designers have used another approach in solving the problem of radiation heat transfer—a set of insulating shields. Surrounding the column is an inner shield of thin copper sheeting. It is attached to the hydrogen reservoir, at the top of the column. Because of copper's high thermal conductivity, the design can provide a shield having the same temperature gradient as the column, top to bottom. Thus, heat radiated from the shield to the column is negligible. Reduction of the heat transferred to this copper shield from outside the column is effected by placing a liquid-nitrogen-cooled shield around the inner shield.[2]

Another major problem encountered in the design and construction of low-temperature equipment involves making proper allowance for coefficients of expansion. For instance, some parts of the pilot plant, including the column, change in length by as much as ½ in. with the 280° K temperature change that takes place in the system. One method of relieving this situation is to solder short sections of bellows to one end of a line. Another way is to use a loop or a couple of bends, in a small diameter line, in order to place a short section of the line at right angles to the principal axis.

As the CEL engineers see it, the only major item causing a change in design between this pilot plant and an industrial unit is the provision made for a supply of pure gaseous hydrogen to the column feed. Since CEL has pure hydrogen available, this pilot plant does not contain a specific purification apparatus. Instead, the bottom product and the distillate are joined together and circulated to the column as feed. Once this column has been charged, a closed circulation system is in force—no new feed is necessary. However, any other designs would have to have an outside source supplying feed continuously to the column.

The designers also recommend traps of absorbents in liquid nitrogen baths or other purifying systems in the feed line to eliminate any plugging of the perforated and screen plates resulting from impurities in the hydrogen gas. Even small impurities can quickly reduce the column's separation efficiency. In fact, if there are sufficient impurities present to clog the plates, the distillation would cease. The authors feel that, even if their recommendations are followed, there is still likely to be an accumulation of impurities for a certain period of time. Occasional shutdowns are probable.

Handling pure hydrogen requires extreme caution. This is as true for hydrogen in liquid form as it is for hydrogen gas. There is always some gas escaping from the liquid, and the liquid represents an extremely concentrated gas supply. The design of the CEL pilot plant incorporates many safety features. An operating pressure of 1.3 atm was selected. This eliminates air leaking into the system and provides a satisfactory temperature difference for heat exchange in the condenser and reboiler. All electrical switches and motors are explosion-proof. All electronic, nonsealed equipment is constantly purged with nitrogen gas. All equipment is grounded. Besides placing the column in a well-ventilated room, the designers have made provisions for either piping the exhaust hydrogen to a gas holder outside the building or to release it through a vent in the roof. Other obvious safety precautions, such as no open flames, methods of reducing high-static potentials and use of nonsparking tools, are maintained. The successful operation of this unit suggests that many other problems in gas separation might be solved by adopting cryogenic techniques.

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[1] *For further details, see* Low-temperature distillation of hydrogen isotopes, *by K. D. Timmerhaus, D. H. Weitzel, and T. M. Flynn, Chem. Eng. Progr.* **54**, *35 (1958).*

[2] *A thorough discussion of low-temperature insulation problems is given in* Cryogenic Engineering, *by R. B. Scott (D. Van Nostrand Co., Inc., New York, N.Y., 1959).*

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Periodicals

Journal of Research of the National Bureau of Standards, Volume **62,** No. 5, May 1959 (RP2950 to RP2956 incl.) 60 cents.

Technical News Bulletin, Volume **43,** No. 5, May 1959. 15 cents. Annual subscription $1.50. 75 cents additional for foreign mailing.

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TECHNICAL NEWS BULLETIN

U.S. DEPARTMENT OF COMMERCE
LEWIS L. STRAUSS, *Secretary*
NATIONAL BUREAU OF STANDARDS
A. V. ASTIN, *Director*

June 1959 **Issued Monthly** **Vol. 43, No. 6**

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Subscription price, domestic $1.50 a year; 75 cents additional for foreign mailing; single copy, 15 cents. Use of funds for printing this publication approved by the Director of the Bureau of the Budget (June 24, 1958).

Contents

	Page
Machine translation of Russian	101
Announcing: New four-part Journal of Research	102
Mechanical degradation of polymers	104
FOSDIC III to assist in 1960 census	106
Thermal stresses in stainless steel box beams	108
New motion picture: Dental X-ray equipment: Alteration for modern radiation hygiene	109
Paper research	110
Symposium on the optical pyrometer temperature scale	114
Low-temperature distillation of hydrogen isotopes	116
Publications of the National Bureau of Standards	119

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Patents

(The following patents have recently been granted on NBS inventions and, except as noted, are assigned to the United States of America as represented by the Secretary of Commerce.)

No. 2,867,382. January 6, 1959. Maneuvering loads accelerometer. Preston R. Weaver (Navy).

No. 2,870,436. January 20, 1959. Electronic analogue-to-digital converter. Milton L. Kuder.

No. 2,871,042. January 27, 1959. Supporting and heat insulating means. Bascom W. Birmingham, Edmund H. Brown, Russell B. Scott, and Peter C. Vander Arend (AEC).

No. 2,871,351. January 27, 1959. Balance detector used in electronic analogue-to-digital converter. Milton L. Kuder.

No. 2,871,356. January 27, 1959. Frequency-stabilized oscillator. Peter G. Sulzer.

No. 2,871,432. January 27, 1959. Automatic tracking proximity gage. Louis A. Marzetta.

No. 2,871,669. February 3, 1959. Radiation shield circulation system for large liquefied gas storage containers. Douglas Mann and John Macinko (Navy).

No. 2,872,538. February 3, 1959. Inertia arming switch. William B. McLean (Navy).

No. 2,872,867. February 10, 1959. Ordnance construction. Robert D. Huntoon (Navy).

No. 2,877,452. March 10, 1959. Telemetering transmitter for a projectile. Allen V. Astin (Navy).

No. 2,879,001. March 24, 1959. High-speed binary adder having simultaneous carry generation. Arnold Weinberger and John L. Smith.

No. 2,879,409. March 24, 1959. Diode amplifier. Arthur W. Holt.

No. 2,879,455. March 24, 1959. Miniature radar subassembly. Robert K–F Scal (Navy).

No. 2,879,657. March 31, 1959. Combination locks. Robert L. Eichberg.

No. 2,880,378. March 31, 1959. Shaped processed circuitry. Clinton O. Lindseth (Navy).